Encapsulated perfumes and/or aromas having a specific release behaviour

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FIELD OF THE INVENTION

The present invention relates to encapsulated aromas and/or perfumes and to processes for their production.

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BACKGROUND OF THE INVENTION

Aromas (flavouring matters) and perfumes are complex liquid mixtures of volatile components. During the production and preparation of aromatized foods and perfumed products, there is the necessity for controlling the release of aromas or perfumes in order to avoid losses.

Especially in the case of water-containing foods which are ultra-heated, protection of the aroma is a technological challenge. In this case, significant aroma losses occur owing to the volatility of the aroma components on heating. In addition, in the case of aroma compositions, due to the differing loss rates of the individual components, shifts in aroma profile can occur. The transfer of the aroma into the liquid during the high-temperature phase in a food processing process must therefore be avoided. For this purpose encapsulation of the aroma is suitable. This aroma capsule should then ideally dissolve in a controlled manner during the cooling phase and thus also release the aroma in a controlled manner.

The application of coatings to particles to establish the solubility behaviour or release behaviour and for protecting encapsulated substances is known. Jackson and Lee, in their review article "Microencapsulation and the Food Industry" (Lebensm.-Wiss.u.-Technol. 24, 289-297 (1991)) enumerate a great number of suitable coating materials, including fats, waxes, hydrocolloids, for example including modified celluloses, and proteins.

WO 97/16078 describes a process only of aroma substances and perfumes which can be encased by a protective skin. As possible casing, inter alia, modified cellulose is also mentioned. The granules themselves are inhomogeneous and comprise a support material and an aroma enclosed in a film-forming agent. The purpose of this application is to produce granules as free as possible from dust. The resultant particles have an irregular shape and an uncontrollable constituent release behaviour.

A reduction in the release rate of encapsulated aromas having a hydrophilic matrix in aqueous systems is customarily achieved by applying coatings of hydrophobic substances, for example fats or waxes, and also of gel-forming proteins or

hydrocolloids. However, for clear aqueous foods, fats or waxes are unsuitable, since visually unacceptable deposits in the food form when they are used.

Although hydrocolloid gels are hydrophilic, that is to say they are colloidally soluble in aqueous systems, the hydration and solubility of the gel increases in many of these systems constantly with increasing temperature, however. Aroma protection is then lowest precisely at high temperatures.

In contrast, certain modified celluloses are distinguished by reversible formation of a solid gel in water at elevated temperatures, which is unique in the hydrocolloid group. The viscosity of these gels increases greatly at high temperatures (above the characteristic flocculation point, that is to say the temperature from which solid, high-viscosity gels are formed), and then decreases again on cooling. The reversibility of gel formation also significantly distinguishes the modified celluloses from the behaviour of protein gels which, although they can also gel at high temperature, their gels do not redissolve on cooling.

This viscosity and temperature behaviour above the flocculation point, which is the inverse of that of other gel systems, and the reversibility of gel formation of certain modified celluloses is termed "reversible thermal gelation" (Edible Films and Coatings: A Review, Food Technology, December 1986, 47-59).

The utilization of the reversible thermogelation of methyl cellulose or hydroxypropyl cellulose in the use as protected matrix for temperature-sensitive substances is known per se.

In WO 92/11084, methyl cellulose is used in a capsule matrix for the sweetener aspartame which is unstable in water-containing media at high temperatures. The stability of the sweetener in bakery products can thus be increased.

WO 98/49910 describes the encapsulation of foodstuffs and other materials, these materials first being encased with a hydrophobic film and then with a layer which has a temperature-dependent reversible solution behaviour. This layer can consist of cellulose derivatives or other polymers. The inner hydrophobic film consists, for example, of fats, paraffin or water. It is also possible that an outer hydrophobic layer is further placed around the polymeric layer having reversible solution behaviour. The encapsulated material can be of variable size and can be present from the food itself or in tablet form. The inner layer can also be present in the encapsulated

material (hybrid system). A disadvantage of this system is the hydrophobic layer, which in an aqueous system deposits on the surface in an unwanted manner.

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SUMMARY OF THE INVENTION

The object of the present invention was, in the production of aromatized, water-containing foods which pass through a heating process, to control effectively the aroma release. The release rate in the cooling phase should be specifically controllable in a time- and temperature-dependent manner up to complete cold water solubility. In addition, the release rates for different aroma components should be approximately equal, in order to prevent unwanted shifting of the flavour profile. Aroma losses are to be decreased by delaying the release at high temperatures.

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DETAILED DESCRIPTION OF THE INVENTION

Encapsulated aromas and/or perfumes have been found which are characterized in that they consist of hydrophilic solid particles in which the aromas and/or perfumes are enclosed and which are encased with or comprise modified cellulose, this having reversible gel formation on temperature increase.

The inventive use of certain modified celluloses for the protection and inversely temperature/time-controlled release of encapsulated aromas and/or perfumes in hot aqueous systems was surprising.

The cellulose for the inventively encapsulated aromas and/or perfumes forms a film which has a high viscosity precisely at high temperatures in aqueous media and is a diffusion barrier for aroma substances. During gradual subsequent cooling, the cellulose gel layer has increased swellability, controllable viscosity decrease as far as complete residue-free solubility. The aroma can, as a result, be released in a time/temperature-dependent manner and linearly. The mode of functioning of the coating (delay rate) can be optimally matched to the respective application requirements.

The modified cellulose forms a casing of the aroma particles and/or perfume particles. The diffusion of the aroma substances or perfumes through the casing layer and thus their release can be controlled via the selection of the cellulose having the specific flocculation point and via the thickness of the casing layer.

The inventive encapsulated aromas and/or perfumes can comprise 1 to 50% by weight, preferably 2 to 20% by weight, particularly preferably 5 to 10% by weight, of modified cellulose. The respective amount of cellulose determines the layer

thickness and controls the release rates for the aromas and/or perfumes, the more slowly the release taking place the higher the cellulose content.

Modified celluloses for the inventive encapsulated aromas and/or perfumes are taken to mean modified celluloses which can form thermally reversible gels. Particular preference is given here to methyl cellulose, hydroxypropyl cellulose, hydroxypropyl methyl cellulose, ethyl methyl cellulose, ethyl cellulose or mixtures thereof.

Reversible thermal gelation cannot occur with all substances which are summarized under the term "modified celluloses". Gels other than the inventive "modified celluloses", for example carboxymethyl cellulose, do not behave in the desired manner.

Hydrophilic aroma particles and/or perfume particles are composed of an aroma mixture and/or a perfume mixture and a hydrophilic support (for example gum arabic or dextrins, such as maltodextrin) which is known per se.

It is also possible to add other substances, for example vitamins, microorganisms, edible acids or colours.

For the present invention it is essential that no further layers are necessary to protect the core.

The invention also relates to a process for producing encapsulated aromas and/or perfumes, in which the aroma particles and/or perfume particles are provided with a coating. This process is characterized in that the coating comprises a modified cellulose with which reversible gelation occurs with temperature increase.

The inventive production process produces encapsulated aromas and/or perfumes of the abovedescribed type having the advantages mentioned there. These encapsulated aromas and/or perfumes can comprise after their manufacture 1 to 50% by weight, preferably 2 to 20% by weight, particularly preferably 5 to 10% by weight, of modified cellulose. Modified celluloses which may be mentioned are in particular methyl cellulose, hydroxypropyl cellulose, hydroxypropyl methyl cellulose, ethyl methyl cellulose, ethyl cellulose or mixtures.

Preferably, the aroma particles and/or perfume particles as serving as core are produced by fluidized-bed spray granulation. The production of these cores is known per se.

The particles have a diameter of 10 to 5000 μm, preferably 200 to 2000 μm.

EP 070 719 describes the production of aroma particles and/or perfume particles in a fluidized bed which is operated batchwise. In this case an aqueous emulsion of the aromas and/or perfumes to be granulated and suitable support materials are sprayed into a fluidized bed which consists of particles vortexed by air. The particles then act as seeds for the formation of granules.

EP 0 163 836 describes an apparatus for producing granules by a continuously operated fluidized bed. The generation of granules and the selected discharge on reaching the desired particle size proceed simultaneously and continuously.

WO 97/16078 describes the production of aroma particles and/or perfume particles in a fluidized-bed rotor granulator which is operated batchwise. Via a rotating base plate, the rotor granulator produces a fluidization of the fluid bed present in it, so that less air is required for its fluidization.

According to the invention preference is given to the continuous mode of producing the particles in an apparatus according to EP 0 163 836.

After the fluidized-bed spray granulation step, by spraying on a solution which comprises water and a modified cellulose, a coating film having uniform defined layer thickness is applied to the aroma particles and/or perfume particles containing the aromas or perfumes encapsulated therein. For this purpose, apparatuses which are known per se, preferably fluidized-bed apparatuses (top-spray coaters, bottom-spray coaters, Wurster coaters) are used.

As solvents for the spray solution there can be used, for example, water or water/ethanol mixtures. The said modified celluloses are made up in the spray solution at a concentration between 0% and 25%, preferably between 1% and 15%. Preferably, for applying coatings, modified celluloses are selected which have a degree of etherification which give only a low viscosity to the spray solution.

The suitable feed air temperatures during coating in the fluidized bed are between 50°C and 140°C. The suitable exhaust air temperatures during coating in the fluidized bed are between 30°C and 100°C.

The layer thickness is 1 to 200 μm, preferably 2 to 100 μm, in particular preferably 5 to 50 μm.

The layer thickness is set by the amount of coating solution sprayed on.

Depending on the application, other substances or else mixtures of substances, for example other hydrocolloids, fats, waxes, sugars or else plasticizers, for example polyethylene glycol or other customary additives, for example food colours, can be added to the spray solution.

In an alternative embodiment of the present invention, the aroma particles and/or perfume particles are encased not by a unitary casing, but by impregnation of the particles with the modified cellulose.

Suitable encapsulated substances are all aroma and/or perfume mixtures which are used in industry, and also individual aroma components and/or perfume components.

The invention in addition relates to a process for enriching foods with aromas or for producing perfumed consumer articles, for example detergents. This process is characterized in that the above described encapsulated aromas and/or perfumes are added to the foods or the consumer articles.

Examples which may be mentioned are infusion bag tea, instant sauce powder, prepared sauce, pasteurized beverages, chewing sweets, wafers, detergents.

Preferably, the foods, during or after the addition of the inventive encapsulated aromas and/or perfumes are heated to a temperature above the flocculation point of the modified cellulose and are then cooled.

By means of the particular release behaviour of the inventive aromas, new qualities of the foods can be achieved. Thus, for example, heating is possible without an excessive aroma loss occurring. During cooling of the foods, vice versa, the desired and defined release of the aromas occurs, which can be controlled in its time course by the type of encapsulation. Since the different individual aroma components are

released at the same rate, and their weight ratio to one another therefore remains constant, no unwanted shifts in aroma profile occur either.

Examples

The invention is described in more detail below with reference to example embodiments with associated figures.

Figure 1 shows the aroma release of encapsulated aromas with and without a coating of modified cellulose.

Figure 2 shows the release of different aroma components.

Example 1

Production of capsules having a release rate of 50% per minute at temperatures above 60°C

A solution of 2.0% by weight of low-viscosity methyl cellulose (viscosity of a 2% strength aqueous solution at 20°C: 400 cP) in water is produced. The flocculation point of this methyl cellulose is above 50°C.

In a fluidized-bed apparatus of the type shown in EP 0 163 836 (having the following features: diameter of gas distributor plate: 225 mm, spray nozzle: two-component nozzle, classifying discharge: zig-zag sifter, filter: internal bag filter) particles which comprise an encapsulated model aroma mixture (consisting of ethyl butyrate: limonene: phenylethyl alcohol, 1:1:1) coated with methyl cellulose. By raising the classifying gas rate to 20 kg/h at 30°C no material is discharged, that is to say coating takes place batchwise. For this operation 480 g of aroma particles are introduced as initial bed charge. The methyl cellulose solution is sprayed into the fluidized-bed granulator at a temperature of 22°C. The temperature of the atomizing gas is 30°C. To fluidize the bed contents, nitrogen is blown in at a rate of 120 kg/h. The inlet temperature of the fluidizing gas is 140°C. The temperature of the exhaust gas is 81°C. Free-flowing granules are obtained. The solid particles are round. The thin, highly uniform methyl cellulose coating is 5% by weight, based on the granule weight.

Example 2

Production of capsules having a release rate of 10% per minute at temperatures above 60°C

A solution of 2.0% by weight of a low-viscosity methyl cellulose (viscosity of a 2% strength aqueous solution at 20°C: 400 cP) in water is produced. The flocculation point of this methyl cellulose is above 50°C.

A coating of methyl cellulose is applied to aroma particles which comprise an encapsulated model aroma mixture (consisting of ethyl butyrate: limonene: phenylethyl alcohol, 1:1:1) as in Example 1, but twice as much casing is applied. For this, coating must be carried out for correspondingly longer.

The methyl cellulose coating is 10% by weight, based on the granule weight.

The functioning of the coating material is described by the diagrams in Figures 1 and 2. Figure 1 shows the delay of aroma release during brewing with hot (boiling) water due to a coating of methyl cellulose (the encapsulated aromas were produced in accordance with Examples 1 and 2).

The time axis on the x-axis extends from 0 to 600 seconds. On the left y-axis is plotted the percentage of aroma released (from 0 to 100%) and on the right y-axis, the respective process temperature (from 0°C to 100°C) can be read off by the associated falling line.

Conventional aroma particles without casing (continuous, steeply increasing line) show rapid release of the aromas. Even after one minute, virtually the entire aroma has been released.

In contrast, the inventive encapsulated aromas release their contents significantly slower. The release rate may be controlled in this case by the methyl cellulose content. At a content of 10% methyl cellulose (dotted curve) it is substantially lower than at a 5% methyl cellulose content (dashed curve).

Figure 2, for the same process, shows the release curves for two different aroma components (dotted and continuous lines). These run almost overlapping. That is to

say that the components are released at the same rate, so that an undesired shift in the flavour profile does not occur.

Example 3

Production of capsules containing strawberry aroma

A solution of 2.0% by weight of a low-viscosity methyl cellulose (viscosity of a 2% strength aqueous solution at 20°C: 400 cP) in water is produced. The flocculation point of this methyl cellulose is above 50°C.

A coating of methyl cellulose is applied to aroma particles which comprise an encapsulated strawberry aroma in a fluidized-bed apparatus of the GPCG 3 type from Glatt having the following features:
Diameter of gas-distributor plate: 150 mm,
Spray nozzle: Two-component nozzle,
Filter: Internal bag filter,
Fluidizing gas inlet temperature: 100°C,
Exhaust air temperature: 60°C,
Atomizing gas temperature: 22°C,
Fluidizing gas rate: 50 kg/h.

The methyl cellulose coating is 10% by weight, based on the granule weight.

4. Application examples

4.1. Tea in infusion bags

To tea in bags are added aroma particles having strawberry aroma encapsulated therein, which are furnished with methyl cellulose coating, and aroma particles without methyl cellulose coating which comprise the same strawberry aroma.

Advantages:
After infusion of the aromatized tea bags, the following are obtained

- both a strong immediate aroma impact which is perceived by odour (orthonasally) and flavour (retronasally),

- and a persistent aromatizing (intensity, aroma profile) of the beverage, which is independent of brewing time over a brewing time of a plurality of minutes.

The losses which occur on infusion with boiling water via transfer of the aroma substances released from the uncoated aroma particles into the water vapour are compensated for in succession with advancing cooling of the tea by gradual and linear release of the aroma from the coated aroma or perfume particles.

4.2. Instant sauce

An instant sauce powder is aromatized with white wine aroma granules which are coated with methyl cellulose. During the preparation, the sauce is heated with water in the pot to above boiling temperature for at least 5 minutes.

Advantages:
The loss of volatile aroma components during heating is reduced. Full aroma action during consumption.

4.3. Prepared sauce

A pasta sauce is aromatized with tomato aroma particles which are coated with methyl cellulose. For preservation purposes, the sauce is heated to 80°C to 100°C for 10 minutes and then cooled in the closed packaging.

Advantages:
The loss of volatile aroma components during heating is reduced. Complete release of aroma does not take place until cooling of the sauce in the closed vessel.

4.4. Pasteurized drink

During the heat-treatment step in a soft drink production process for preservation, granules containing encapsulated ethyl butyrate are added which are coated with a film of methyl cellulose.

Advantages:
Improvement of the aroma profile by protecting the volatile ethyl butyrate during the heating step and subsequent complete release of the ethyl butyrate during the cooling process in the closed vessel. The coating leaves no residue behind in the final drink.

4.5 Chewing sweets

Red-dyed aroma granules containing raspberry aroma encapsulated therein and methyl cellulose coating is added prior to shaping at 1% into the hot (120°C) chewing sweet mass which comprises sucrose, water, glucose syrup, fat, fondant, gelatin, citric acid and an emulsifier, and the mixture is then cooled and aerated.

Advantages:

- The granules do not dissolve during the production process, so that a visual effect can be achieved via the conspicuous granules in the end product.

- Low aroma losses occur during the processing operation.

The aroma is present in the matrix localized at few points and does not migrate. As a result, a special sensory effect is achieved (hot spots). The surrounding chewing sweet mass can be aromatized with another liquid aroma, whereby a double sensory effect can be achieved.

4.6. Detergent

Granules which comprise an encapsulated perfume combination (lily of the valley fragrance) and which is furnished with a coating of modified cellulose is used to perfume washing powder.

Advantage:
The perfume dose in the detergent can be reduced. Loss of perfuming during washing of clothes via leaching with the washing water is minimized, since the aroma particles adhere to the clothing fibres. The encapsulated perfume is protected in particular at high washing temperatures.

4.7. Ice cream wafers

Yellow-dyed aroma granules having lemon aroma encapsulated therein and a 5% strength methyl cellulose coating are added at a dose of 2% by weight to a dough mixture for manufacturing ice cream wafers. This dough mixture consists of water (45%), wheat flour (35%), sucrose (15%), ground nut oil, lecithin, salt. The dough

containing the aroma granules is then poured out thinly onto a 250°C wafer iron and baked for 1.5 minutes. The wafers are then rolled up to form cones.

Advantages over uncoated aroma granules:
The aroma granules are retained during the baking process and only small losses of the encapsulated volatile lemon aroma occur. The aroma is not released until during consumption, mechanically by chewing.

Due to the localization of the aroma at individual points in the wafer, a special sensory effect can be achieved.

A visual effect can be achieved by the retention of the conspicuously dyed granules.